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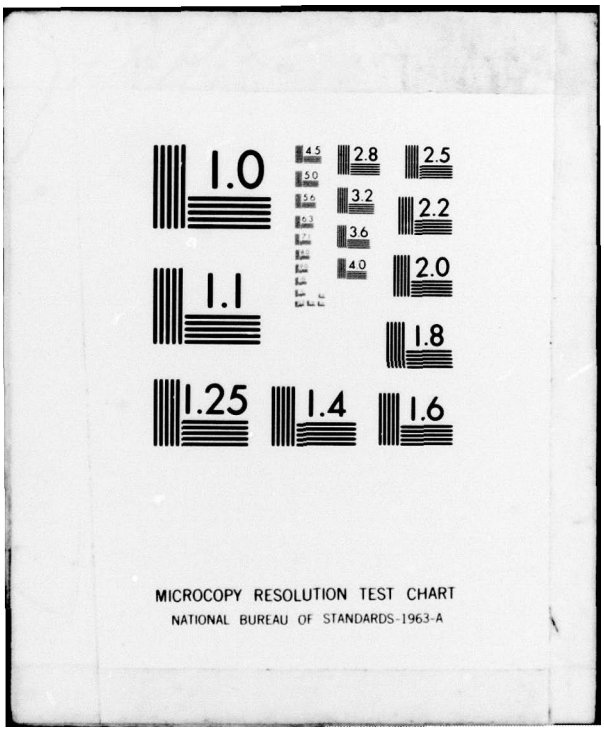
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Supersaturated Aluminum Alloy Powders

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20. Abstract Four aluminum alloys were prepered as powders by the RSR Process. The compositions (in weight percents)were : (1) AL-7Fe-0.5Cr. (2) AL-7Fe-1.0Cr. (3) AL-7Fe-1.5Cr. (4) AL-7Fe-1.0Cr-0.2Ti-0.2v-0.2Zr. The alloys were compacted and extruded at 750 F with an extrusion ratio or 39.3:1. Microstructural investigations of both the powders and the bulk extrusion were made.		

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This report has been reviewed by the Information Office (EOARD/CM) and is releasable to the National Technical Information Service (NTIS). At NTIS it will be releasable to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

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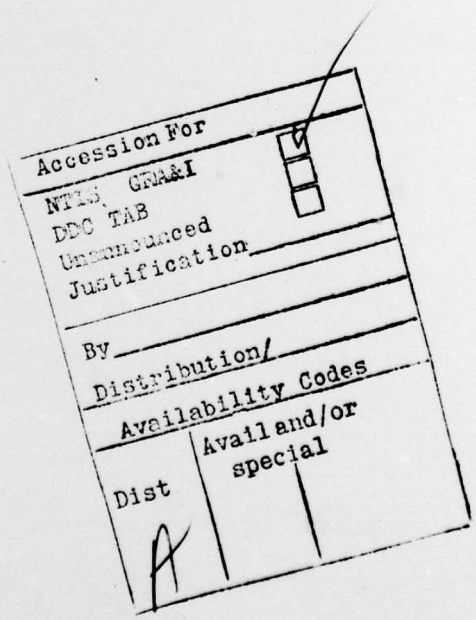


1. General

The first year of this study was divided into two periods. The first period was dedicated for the development of a technique for the making of transmission electron microscopy specimens. The results and recommendations of conditions for making thin foils are given in this report.

In the second period, four Aluminum alloy powders were prepared for this research. The powders ordered by Dr. L.R. Bidwell from P&W Aircraft later compacted, evacuated and extruded by the AFML Experimental Metals Processing Laboratory at W.P.A.F.B.

The first investigations on the powders and on the extruded bars are reported here.

Acknowledgement

I would like to than Dr. L.R. Bidwell and Mr. W.M. Griffith of the AFML/LLS for consulting for the project.

I also wish to thank Mr. A.M. Adair of the AFML/LLM and the Westinghouse contract group headed by Mr. I. Martarell for their cooperation in the processing of the powders, and P.& W.A. for making the powders.

2. The Scientific Work

a. T.E.M. thin foils preparation.

Thin foils of supersaturated aluminum alloy powders held in nickel matrix were prepared for TEM study.

The preparation of the foils included the following stages:

1. Electrolytic nickel deposition simultaneously with aluminum alloy powder.
2. Thinning by electropolishing.

The following is a summarized description of these stages:

1. Nickel sulphamate bath was used for the electrodeposition of the nickel.

The composition of the solution is as follows:

Nickel sulphamate	-	$Ni(NH_2SO_3)_2 \cdot 4H_2O$	-	600 gr/liter
Nickel Chloride	-	$NiCl_2 \cdot 6H_2O$	-	4.4 gr/liter
Boric Acid	-	H_3BO_3	-	40 gr/liter
Distilled water			-	Balance
Antipitting agent			-	1 gr/liter

The bath is kept at - PH-3.5÷4.5 for minimum internal stress formation.

The conditions in which the coating is made are

Temperature - 50°C

Current density - $5 \frac{\text{Amp}}{\text{decimeter}^2}$

Continuous mild stirring of the solution is necessary.

The anode is made of ultrapure nickel which represents excellent efficiency.

The Cathode is made of austenitic stainless steel to which the nickel does not adhere.

The powder to be embedded in the nickel is suspended in the solution by the the stirring operation and is collected onto the horizontal cathode by gravitation. During this process, the powder is coated and held by the Nickel foil formed. When the coating reaches the thickness of about 200 μm the process is stopped and the powder containing nickel foil is peeled off the cathode.

2. The thinning process developed for the making of TEM specimens starts with the punching of discs, 3 mm in diameter from the nickel foil.

   The roughness of the surface of the discs is smoothed by mechanical grinding and a two stage electropolishing process is carried out.

Electropolishing (stage I). The electrolyte found best for electropolishing the composite coating Ni-AL, is sulphuric acid H_2SO_4 - 6% in methanol. This polishing stage is done in a jet apparatus designed and built for the project. Conditions of the electropolishing process are as follows:

Jet diameter - 1.5 mm
D.C. voltage - 100 volts
Temperature - Room
Distance between cathode (austenitic stainless steel) and specimen - 6 mm
The disc is mounted on a grid and minimum jet stream without breaking into drops is used (flow gravitation).
Both sides are thus electropolished in a period of about 30 seconds.
When the thickness at the center of the concaved disc reaches 50 μm the process is stopped and the specimen is subjected to the second electropolishing stage.

Electropolishing (stage II). This stage is carried out slowly to ensure proper inspection of the first stage of perforation and even polishing of both surfaces of the disc.

Electropolishing is done using the same electrolyte as in stage I and the window technique is used. Other conditions are as follows:

D.C. voltage	-	15 volts
Temperature	-	-40°C or less
Cathode	-	Austenitic stainless steel

Good thin foils for TEM study are obtained after 15 ÷ 30 min.

This technique has been tested at the Technion and at the AFML/LLS (thinning by ion beam bombardment) and was found satisfactory.

b. The powders

RSR (Rapid Solidification Rate) process of P&W Aircraft was used to make the four powders. The composition of the powders are as follows: (numbers indicate weight percent)

#1 AL-7Fe-1.0 Cr - 0.2 V-0.2 Zr-0.2 Ti

#2 AL-7Fe-1.0 Cr

#3 AL-7Fe-0.5 Cr

#4 AL-7Fe-1.5 Cr

Alloys were prepared by P.W.A. according to the composition specification ordered by D. Shechtman through Dr. L. Bidwell of the AFML/LLM.
The size and shape of the powder particles were examined in the scanning electron microscope as will be described later in the report. Transmission electron microscopy of the powders as well as X-ray analysis of the structure STEM analysis of the precipitates particles are being done now and will be reported in a later report.

c. The processing of the powders.

The following steps were taken to extrude the powders.

(a) The powders were partially consolidated in a rubber bladder to a cylinder which size matched the dimensions of the can. During this process one of the bladders tore off and the powder AL-7Fe-1.5Cr was contaminated with oil. Nevertheless, this powder was treated later on like the other powders, even though we do not expect good properties from this contaminated extruded rod. A quantity of about half a pound was left from each powder, and was divided into two equal parts between Dr. Bidwells group at the AFML/LLS and the laboratory at the Technion.

(b) The compacted cylinders were inserted into the cans (made of 6061 Alloy) and the cans were sealed except for an evacuation tube opening. At this stage the cans were evacuated and heated to 900°C (482°C)

for degassing. The degassing temperature was designed to reach the highest expected extrusion temperature to eliminate hydrogen blistering in the final extrusion product. The degassing process stops after no signs of gas were observed on the pressure guage. The tubes were sealed at this stage and cans were ready for extrusion.

(c) Extrusions of the billets were made at a ratio of 39.3 to 1. The die was preheated to 500°F and the billet to 750°F. The extruded bars had a length of about 150", and were 1/2" in diameter. About half of each of the four bars was left at the AFML/LLS, and the rest taken to the Technion.

d. Microstructure

Optical, scanning electron microscopy and transmission electron microscopy were utilized to study the powders and the extruded bars, prior to heat treatments. The powders as photographed in the scanning electron microscopy are shown in fig. 1.

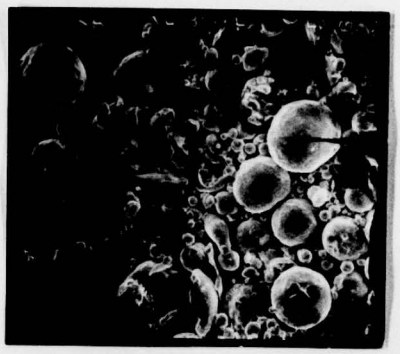
Note that the powder particles vary in size and range between 5 to 130 μm and have irregular shapes. These two features are desirable for compaction purposes. Some of the particles are shaped like flakes, probaly due to impact while in liquid state. Another phenomenon observed in all the powders is shown in fig. 2. This shape is thought to form when a molten drop impacts at a solid particle thus smearing on its surface.

T.E.M. observations of the consolidated material were carried out in a Jeol 100B microscope with an accelerating voltage of 100 kv.

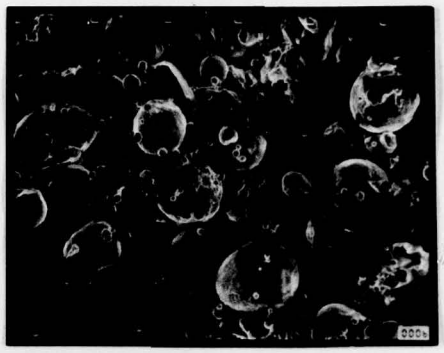
Specimens of extrusion 1 were thinned by electropolishing using the following conditions:

Electrolyte:	Methyl alcohol	-	950 cc
	HNO_3	-	15 ml
	Perchloric acid	-	50 ml
Stage #1	Voltage	-	100 volt
	Temperature	-	R.T.
	Technique	-	jet polishing
	Final thickness	-	50µm
Stage #2	Voltage	-	15 volt
	Temperature	-	-50°C
	Technique	-	window
	Final thickness	-	0

The extruded rod was found to be recrystallised, having average grain size of about 1 µm fig. 3 . A high density of homogeneous precipitation is observed fig. 4 . Precipitate size is about 100 to 200 nm having various shapes. At this stage we have not identified the various kinds of precipitates but X-ray and STEM work are being carried out, aimed at identifying the precipitates crystallography and composition.

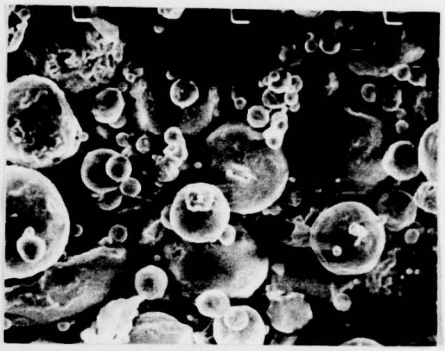


Powder No.1 x100



Powder No.2 x100

Powder No.3 x100



Powder No.4 x230

Fig.1. SEM Micrographs of the four powders.

Fig.2. Shape formed on impact of a molten drop at a solid particle. x800

Fig.3. Partially recrystallized extrusion No.1
as extruded TEM x40,000

Fig.4. Precipitation in extrusion No.1
as extruded TEM x40,000